

7. SAMPLING, TREATMENT, AND DISPOSAL

This section describes the sampling, treatment, and disposal of cylinders removed from CPP-84. Soil sampling at CPP-84 and CPP-94 is also required to verify that COPCs have been removed by the remedial action. Backfill and regrading operations will follow confirmation of contaminant removal.

Sampling of all cylinders will likely be required due to the uncertainty of using external characteristics to define cylinder contents. Toxic gas cylinders are not expected to be recovered from CPP-84 and cylinder contents can either be thermally oxidized (for flammable gases) or vented to the atmosphere (for inert gases). The disposal of cylinders after treatment is dependent on cylinder contents. Prior to sending wastes to an off-Site (off of the INEEL) storage, treatment, or disposal facility, a suitable assessment will be performed in accordance with 40 CFR 300.440.

7.1 Objectives and Approach

The objective of cylinder sampling is to determine the contents of each of the cylinders. This is a fundamental requirement since knowledge of cylinder content is required prior to treatment (venting or flaring) or off-Site transportation. CGA P-22 offers the following guidance:

“Any inconsistency, question, or lack of knowledge about the cylinder is cause for requiring positive identification through sampling and analysis . . . Equipment used for sampling cylinder contents should be rated for the maximum pressure which could be in the cylinder with a suitable safety factor applied for potentially over-pressurized containers.”

After cylinder contents are identified using an onsite laboratory, treatment methods can be determined. Figure 7-1 provides a cylinder sampling and treatment flow chart that summarizes sampling and treatment options. These options include onsite operations (venting and flaring) and off-Site treatment.

Soil data will be collected at CPP-84 and CPP-94 at the conclusion of cylinder removal activities. The purpose of this data collection effort is to provide a characterization of the excavation bottom. Soil samples will be collected and analyzed for COPCs at an off-Site laboratory. Details of this soil sampling are provided in the *Preliminary Characterization Plan for OU 3-13, Group 6, RD/RA Buried Gas Cylinders Sites: CPP-84 and CPP-94* (DOE-ID 2000a) (Attachment 1).

7.2 Cylinder Sampling

Cylinder sampling techniques are based solely on cylinder and valve integrity. For cylinders with operable valves, a remotely operated system, the VSS will be used. This system allows the operator to remotely view the sampling operation using video equipment. For cylinders that are in poor condition or with inoperable valves, the CRV will be used. The CRV is a remotely operated, pressure-rated vessel that is housed within in a secondary containment chamber for the containment of fugitive gases. The cylinder is pierced within the CRV, allowing the contents of the cylinder to be sampled and analyzed.

7.2.1 Valve Sampling Station (VSS)

The VSS is designed to provide remote valve sampling capabilities for compressed gas cylinders with operable valves. The unit consists of a 54-in. x 54-in. x 84-in. containment structure, which is constructed of ¼-in.-thick steel plate which is trailer- or vehicle-mounted. Other components of the system include a secondary containment structure, remote valve opening capabilities, video equipment, and emergency treatment capabilities. Figure 7-2 provides a schematic of the VSS. Complete process engineering diagrams and operating procedures will be available at the job site.

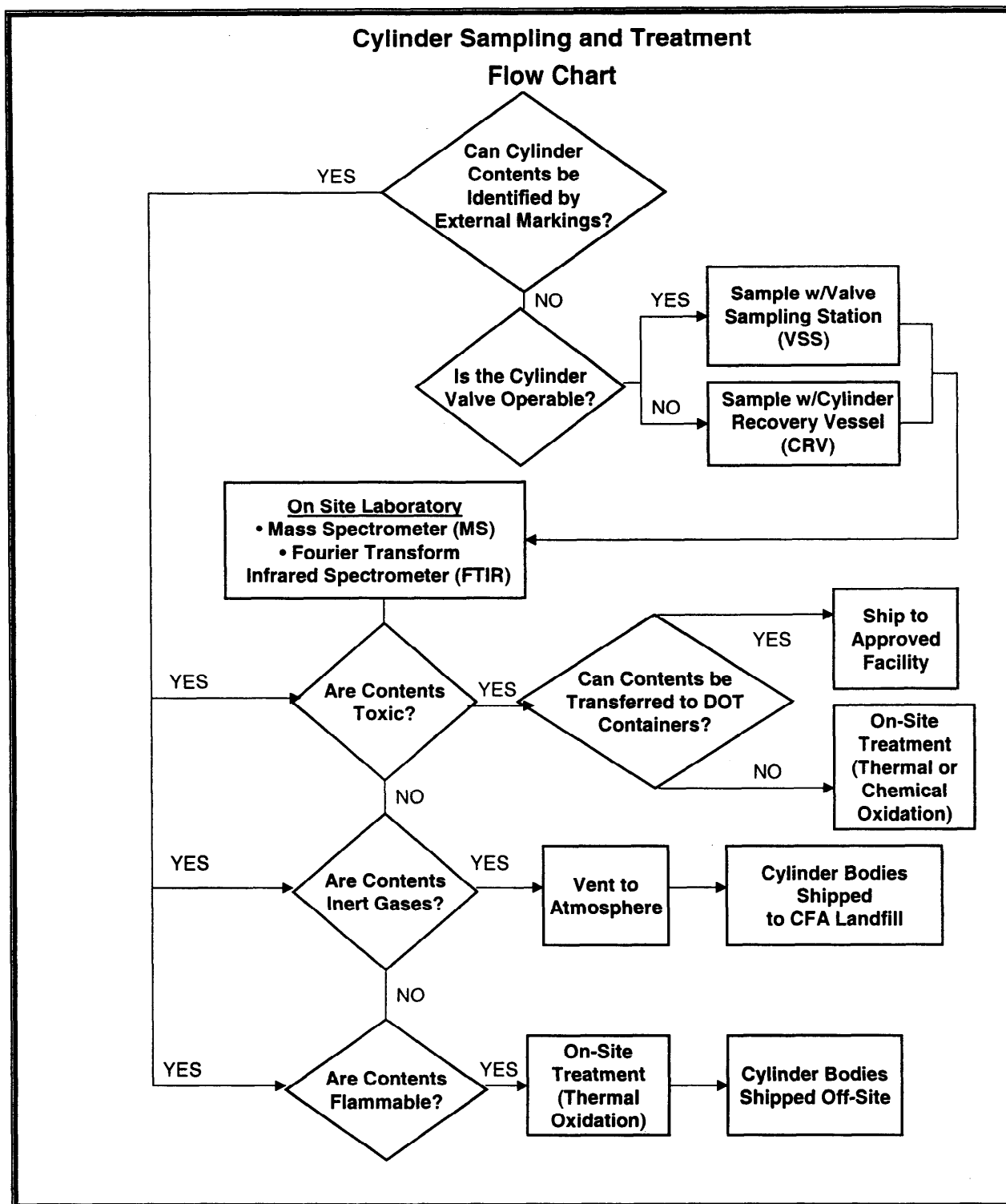


Figure 7-1. Cylinder sampling and treatment flow chart.

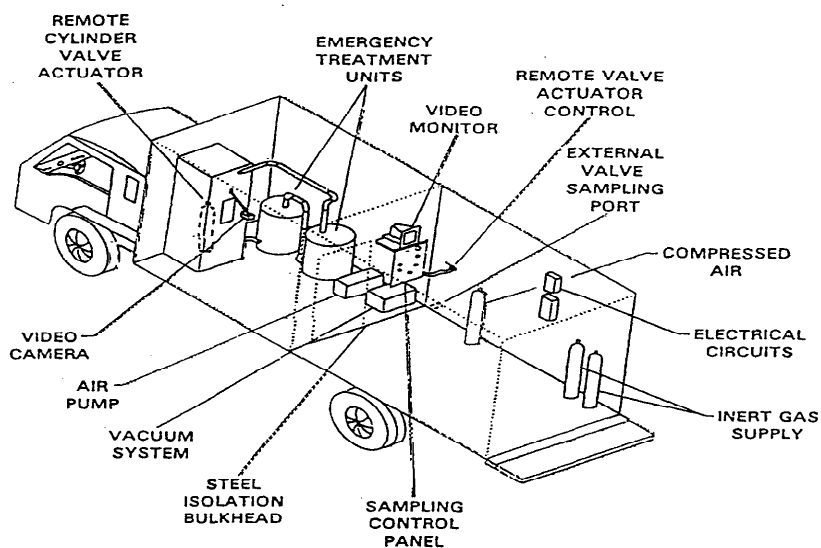


Figure 7-2. Schematic of the VSS.

7.2.2 Cylinder Recovery Vessel (CRV)

The CRV is designed to sample liquids and gases in cylinders that cannot be accessed through the valve or because the cylinder is deteriorated and/or in unstable condition. The CRV provides a remotely operated system to release typical cylinder contents into a controlled, contained environment. After sampling and analysis of cylinder contents, the material can be transferred to a new container or disposed through various treatment processes. Figures 7-3 and 7-4 provide a photograph and a schematic of the CRV.

The CRV door is hydraulically operated and contains two o-rings that provide a vapor-tight seal to the outside environment. As the cylinders are loaded into the unit and supported on a specially designed equipment rack, a spring mechanism holds the cylinder in position in the center of the vessel. The cylinder is accessed by either shooting a nitrogen-powered steel projectile through the cylinder wall or using a tapping device. Once the cylinder has been pierced, sampling is completed through a sample port for analysis at the onsite laboratory. Complete process engineering diagrams and operating procedures will be available at the job site.

7.2.3 Sample Analysis

Analysis of cylinder contents will be performed by two methods: (1) FTIR or (2) MS. The infrared spectrum contains characteristics that permit identification of functional groups, or "working parts" of molecules. Through the use of an interferometer, infrared wavelengths are passed through a sample simultaneously. A laser is used to align the optics used in the process.

The FTIR will be used to qualitatively identify cylinder contents through a comparison of spectra with library references. Spectral libraries are maintained with the laboratory computer. Computer libraries are supplemented by several standard hard-copy references. The FTIR is applicable for all but elemental gases (oxygen, nitrogen, etc.). For elemental gases, the MS is the preferred method of analysis.

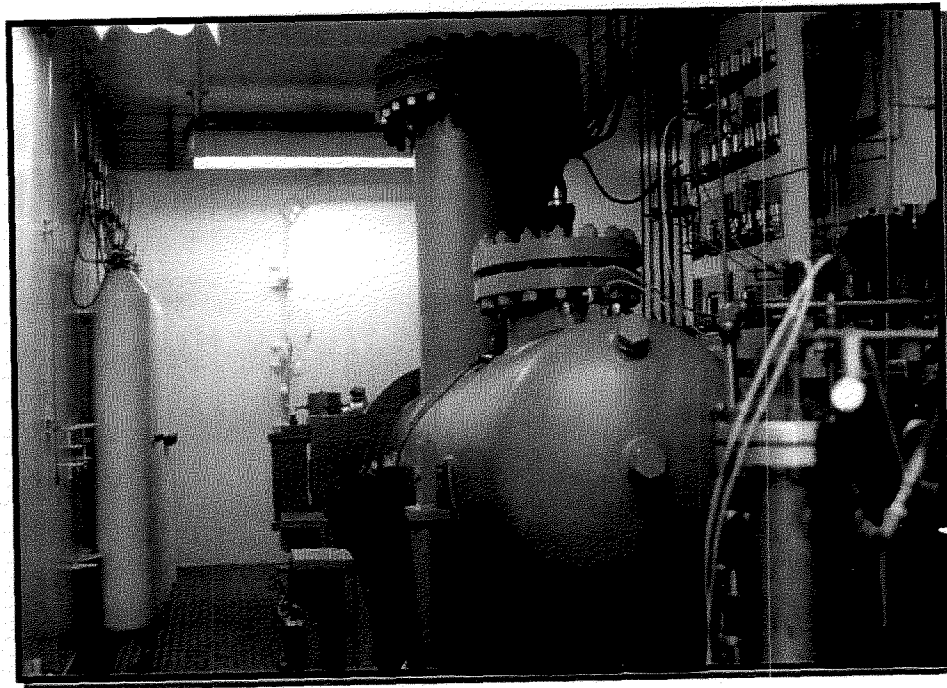


Figure 7-3. Photograph of CRV.

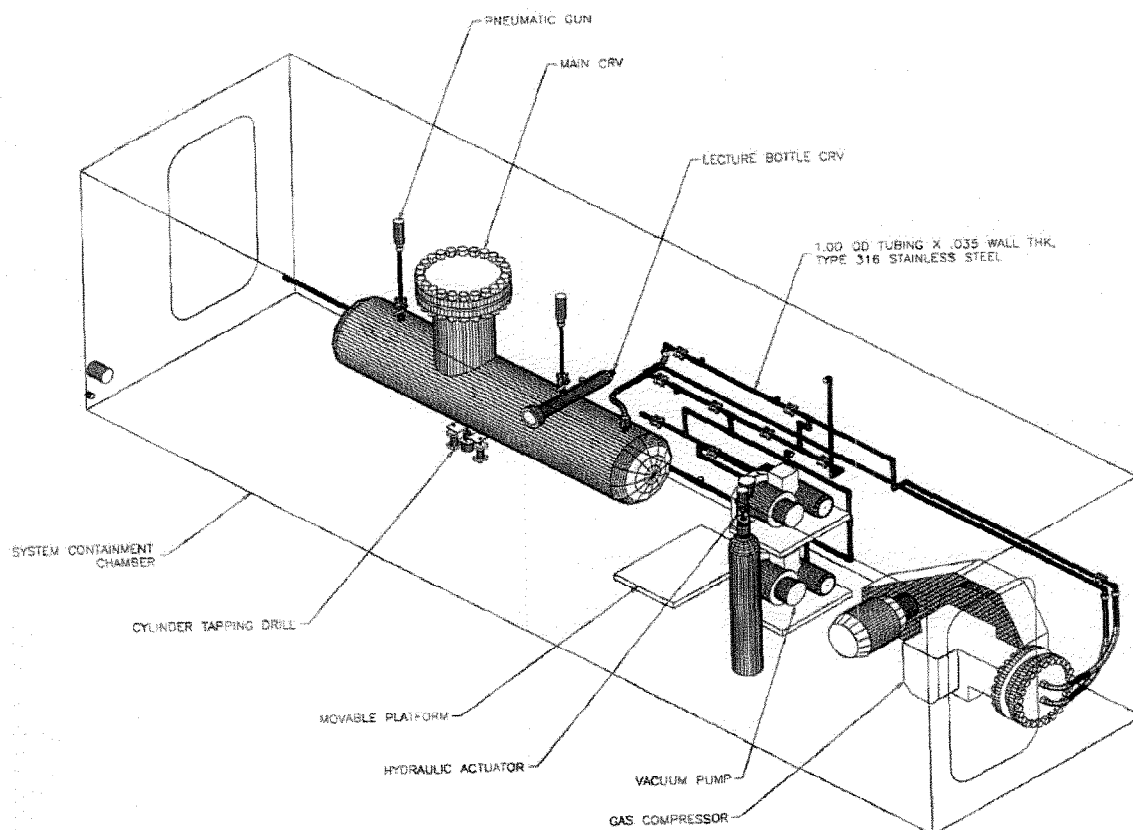


Figure 7-4. Schematic of CRV.

The MS is a vacuum analyzer, which will measure total and partial pressures. The analyzer is a quadrupole mass spectrometer that is capable of separating ions formed in an electron impact source according to the mass-to-charge ratio. The signal collector is either a Faraday Cup or Secondary Emission Multiplier.

The QA/QC procedures used for the instruments will be used only on qualitative analyses. Sample folders will be developed for each cylinder processed. Both the FTIR and MS are computer-supported and calibration/analyses data will be stored at the site on diskette. A hard copy of the spectrum will also be obtained and stored with project records. General items provided in the sample folders include sample identification number, date and time of analysis, and cylinder inspection log reference. Table 7-1 provides a summary of the specific calibration and operation data for each instrument.

7.2.4 Fourier Transform Infrared Spectrometer

Mathematical manipulation of the interference data allows identifying characteristics to be developed. Software used with the FTIR facilitates matching of spectra against various libraries of more than 15,000 compounds. The spectrum is mathematically converted to absorbance. The absorbance spectrum is then compared to those contained in the reference libraries.

Several FTIR spectral libraries are maintained on the laboratory computer. These include the 3,000 compound EPA Vapor Phase Library, a gas library, and Aldrich's library of compounds (30,000 compounds). Database searches are conducted with Sprouse Scientific Search software. The computer libraries are supplemented by several standard reference books.

The FTIR will be checked daily for operability in accordance with the equipment manual. A polystyrene film is used for calibration and checked against three principal peaks (702, 1602, 3025 cm^{-1}). The QA/QC plan for the mobile laboratory specifies the frequency and limits of calibration.

7.2.5 Mass Spectrometer (MS)

The MS to be used at the site can identify compounds between 1 and 300 a.m.u. This will cover the range of elemental gases that are not identifiable with the FTIR. It will be calibrated at the beginning and end of each day using bromofluorobenzene (BFB) as a standard.

Table 7-1. Specific calibration and operation data.

| FTIR | MS |
|---|------------------------------------|
| Wavelength tune spectrum | Argon tune |
| Wavelength tune check (polystyrene) | BFB start analog plot and form |
| Spectrum and form | |
| Plots of blanks and sample spectra | BFB end analog plot and form |
| Wavelength and tune checks (end of day) spectrum and form | Intpretation and reference spectra |
| Intpretation and computer match | Analog plots of blanks and sample |

Mass spectrums for each sample are shown as peaks. A hard copy and electronic copy are provided for each analysis. The spectrum is then compared with library spectra for aiding identification. MS data are compared to a proprietary library of compiled cracking patterns (0 to 200 a.m.u.) The library contains both mass data and peak intensities for approximately 50,000 compounds.

Spectra selected from the search are compared with known chemical characteristics of the sample gas. In some cases, physical observations obtained during sampling can confirm the interpretation. Both MS and FTIR spectra are contained in the Chemical Information System database. This computerized database can be accessed on-line.

7.3 Treatment of Cylinder Contents

Analysis will confirm whether the gases contained in the cylinders are common industrial gases typically associated with construction operations. Following laboratory confirmation of cylinder contents, the industrial gases will be disposed of by either controlled venting or flaring. These treatment processes will render the cylinders as being empty in accordance with the definition of 40 CFR 261.7(b)(2). If cylinders contain gases other than the expected construction gases, they will either be treated on-site or be shipped to an appropriate off-site facility.

7.3.1 Treatment of Anticipated Gases

Controlled venting of the contents is an option suitable for inert or innocuous materials. Typically these are common components of air. These atmospheric gases include air, argon, carbon dioxide, oxygen, helium, and nitrogen. The primary hazard associated with these gases is concentration in a confined area. Controlled and monitored venting will permit these to be released without further processing.

Acetylene is the only anticipated flammable gas to be encountered during the removal action. Cylinders containing acetylene will be treated via thermal oxidation. The feed rate of the acetylene gas will be monitored as well as the surrounding areas to ensure that excessive pressure is not built up in the system and no explosive atmospheres are created.

The techniques used to vent inert gases or thermal oxidize the acetylene are essentially the same and use the same equipment. Gases will be vented through an established flare stack. The flare stack consists of lines (1-in. diameter or less) connecting the sampling device with an industrial burner and pilot light. A flash arrestor will be installed in the line prior to the burner to prevent flushing back to the ignition source. The stack will be located at least 50 ft from any source of ignition other than the associated pilot flame. A clear zone of the same radius will be maintained during the processing. The flame will be fueled from a propane cylinder located outside of the clear zone. A fire watch will be maintained from the start of venting until 30 min after the flaring is concluded. The clear zone shall be delineated to prevent unauthorized personnel from entering the area while a flame is present or while the burner is still hot. Perimeter monitoring will occur for LEL, percent oxygen, and SO₂. The venting and flaring requires evacuation and purge cycles to assure that residual gases are removed from each container. General operating procedures are as follows:

1. Identify personnel
 - Trained operator to feed the gas
 - Trained watch to observe the burner/outlet

2. Conduct preoperational checks
 - Pressure test and inspect the feed and burner system
 - Compare each cylinder to the analytical results to verify contents
 - Inspect the fuel supply system for secure fittings, condition, and proper pressure regulator adjustment
3. Establish safety systems
 - Establish radio communications between operator and watch
 - Provide fire extinguisher at watch location
 - Exclude flammable materials from the area
4. Conduct treatment operations
 - Notify watch the feeding will commence
 - Open valves to feed gas
 - Monitor system pressure to ensure it is maintained within established limits
 - Continue feed until cylinder pressure reaches atmospheric
 - Evacuate feed lines
 - Purge system with nitrogen
5. Conduct post-treatment activities
 - Continue watch for 30 min after completion of treatment
 - Dispose of empty cylinders in accordance with the *Waste Management Plan*.

7.3.2 Treatment of Non-Anticipated Gases

Although it is unlikely that other gases will be encountered during this project, treatment options for nonanticipated gases require identification. If other gases are retrieved, they will be managed and treated on a case-by-case basis depending on the characteristics of the gas. These gases may be treated onsite or sent to an appropriate off-Site facility for treatment and disposal.

Depending on the gas type, onsite treatment may be conducted using the venting or thermal oxidation described above, or by more complex catalytic or chemical oxidation technologies. Due to the large number of gases, it is not feasible to describe a detailed treatment process for every gas type. Table 7-2 describes suitable methods for managing a variety of common gas types that could be encountered.

Table 7-2. Treatment methods for non-anticipated compressed gases. (Source: DLAR 4145.25, 1/90)

| Gas | Treatment |
|-------------------------|---|
| Anhydrous ammonia | Convert to ammonium nitrate by passing vapors into nitric acid solution |
| Chlorine | Neutralize by passing vapors into 18–20 % sodium hydroxide solution |
| Dimethylamine | Neutralize by passing vapors into a nitric acid solution |
| Ethyl chloride | Neutralize by passing vapors into sodium hydroxide solution |
| Hydrogen chloride | Neutralize by passing vapors into sodium hydroxide solution |
| Hydrogen sulfide | Neutralize by passing vapors into sodium hydroxide solution |
| Methyl bromide | Neutralize by passing vapors into sodium hydroxide solution |
| Methyl chloride | Neutralize by passing vapors into sodium hydroxide solution |
| Liquified petroleum gas | Thermal oxidation |
| Phosgene | Neutralize by passing vapors into sodium hydroxide solution |
| Sulfur dioxide | Neutralize by passing vapors into sodium hydroxide solution |

7.4 Post-Removal Characterization Activities

Post-removal characterization activities at CPP-84 and CPP-94 consist of (1) soil sampling to estimate the average concentrations of COPCs in the excavation and, if needed, the spoil pile and (2) a confirmation magnetic field survey. Based on the DQOs of this project, a simple random sampling design (utilizing composite samples) will be used for locating sampling locations (Table 3-2). The design described in this section allows for estimating the variability (standard deviation) of the COPCs (if present) and also allows for comparing the COPCs against action levels using a student's *t*-test. The option to collect additional biased samples will be reserved if evidence (such as discoloration, staining, textural differences, odors) indicates contaminants could be present in an area that might otherwise be missed (e.g., spoil pile, excavation portions not containing cylinders). The following statistical parameters, sample frequency, and sampling techniques described in this section were established using EPA guidance (EPA 1989, EPA 1991, EPA 1992a, EPA 1992b, EPA 1994, and EPA 1996):

- Confidence Level: 80%
- Minimum Detectable Difference: 30%
- Power: 90%
- Coefficient of Variation: 30%
- Five samples (plus 1 duplicate) from CPP-84 excavation and, if needed, samples from the spoil pile
- Five samples (plus 1 duplicate) from CPP-94 excavation and, if needed, samples from the spoil pile.

7.4.1 Sampling Design for Excavated Areas

7.4.1.1 Establish Sampling Grid. Using maps, the excavated areas will be divided into grids. Grid cell sizes will be determined in the field based on the size and distribution of the cylinder area. The following procedures will establish the sampling grid:

1. Measure the horizontal (x - y) extent of cylinder distribution in square feet (ft^2). Assess the distribution of the cylinders on the horizontal plane. If there are significant gaps or distances between cylinders that would cause the sampling of > 1 grid cell that did not contain a cylinder (and there is no visual evidence of contamination), then do not include that area in the calculation of cylinder distribution. The purpose is not to include large portions of the excavation in which no cylinders were present.
2. If the area of cylinder distribution is $< 750 \text{ ft}^2$, then divide the site into a minimum of 30 equally sized grid cells.
3. If the area of the cylinder distribution is $> 750 \text{ ft}^2$, then establish grid sizes of 25 ft^2 (e.g., $5 \times 5 \text{ ft}$).
4. After establishing the grid size and dividing the site into grid cells, assign a unique two digit number (01, 02...30) to each grid (if more than 99 grids are required, use a three-digit number).
5. Select five grid cells for sampling using a random number generator or table.
6. Document all activities, drawing, calculations, and measurements in the field logbook.

7.4.1.2 Collect Bulk Soil Samples. At each sampling grid, bulk quantities of soil will first be collected. Each sample will be a composite of five aliquots (i.e. sub samples, portions) using a '5 on die' design (see Figure 7-5). The following procedures describe how to collect the bulk soil samples (Pitard 1989):

1. At each composite location within a grid, use a disposable/dedicated spoon to collect surface samples (using the bottom of the excavation as the revised 0 datum point) from 0 to 2.5 cm (1 in.) of soil. Place the soil into a large sealable plastic bag (similar to Ziploc™) and label appropriately.

Note: For this project, soil is defined as particles $\leq 2 \text{ mm}$ in diameter and absent of gross size organic materials. If sieving is required, pass the soil through a pre-cleaned #10 (2 mm) sieve (#9 Tyler equivalent).

2. For volatile organic compound (VOC) samples, place sample aliquots directly into the appropriate sample jar and fill to minimize headspace. The priority for minimizing the amount of time the soil is exposed to air outweighs the additional rigor on optimizing sample representativeness.
3. Estimate the amount of soil needed from each aliquot so that the bulk volume collected at each grid is about 50% more than the amount needed for filling analytical sample jars.

Note: For the duplicate sample, collect enough sample material to fill two sets of analytical sample jars.

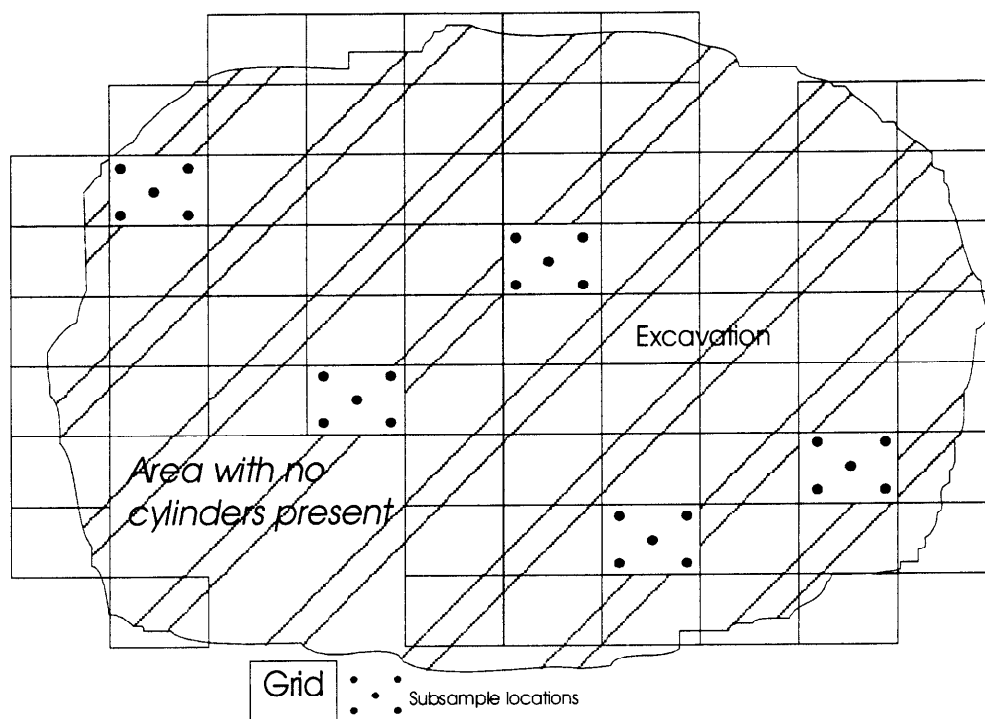


Figure 7-5. Hypothetical sampling grid.

4. Label the sealable plastic bags with the date, location, and sample number using an indelible marker, and keep the sample securely stored at 4°C until ready for sample processing.

7.4.1.3 Sample Processing. A one-dimensional incremental delimitation method will be used to process the bulk samples into individual analytical samples. The following describes the how to process the bulk soil samples:

1. Prepare the appropriate number and types of empty sample jars as required. Remember to prepare additional jars for the duplicate sample.
2. For each sample, line the bottom of a flat-bottom tray (e.g., cookie sheet, food tray) with new aluminum foil. Transfer the soil from a sealable plastic bag onto the tray and shape the soil into a flat rectangular pile with uniform thickness.
3. Using a disposable/dedicated flat-bottom spatula, collect increments across the soil pile and place them into the sample jars in a sequential fashion. Ensure that each spatula scoop encompasses the entire profile of the soil pile (i.e., include soil fines).
4. Reshape the soil pile as necessary to maintain uniformity. Use at least 25 to 30 increments to fill each jar. Continue until all sample jars are about 90% full.

Note: *Because VOC samples are already collected directly into their sample jars in the field, no further sample processing is required.*

5. Ensure all jars are labeled with all the necessary information for shipment to the laboratory. Securely maintain the sample at 4°C until ready for shipment to the analytical facility.

7.5 Disposal

The non-acetylene RCRA empty cylinders (40 CFR 261.7 [a] [1] and [b] [1]) meeting the *INEEL Reusable Property, Recyclable Materials, and Waste Acceptance Criteria* (RRWAC)(DOE-ID 1999b) for industrial waste will be disposed at the INEEL Landfill Complex. These cylinders will be rendered useless through cutting, drilling, and/or valve removal. Acetylene cylinders are constructed with a porous filler (usually asbestos) and a solvent (acetone) to provide for safe operations. Due to environmental and waste management concerns regarding these substances, after the oxidation of the cylinder contents, the cylinder bodies will be transported to an off-Site disposal facility.

7.6 Backfilling

When verification sample results indicate that site contamination levels are within acceptable limits and the ER department approves the results, the excavated areas will be backfilled. Clean fill material staged near the excavation will be placed in 8-in. loose lifts and compacted to approximately 90% of maximum dry density of the soil with heavy equipment (e.g., bulldozer and/or trackhoe). Figure 7-6 shows a backfilling operation. In the event that heavy equipment is too large to effectively provide compaction, smaller compaction equipment (such as walk-behind roller compactors, mechanical tampers, or vibratory plates) will be used. It may be necessary to add clean water at times to reach the necessary compaction.

All excavations will be surveyed prior to backfilling and restoration. Backfill material will support revegetation. When the original grade is restored, the disturbed areas will be revegetated with native species according to *Guidelines for Revegetation of Disturbed Sites at the INEL* (Anderson and Shumar 1989).

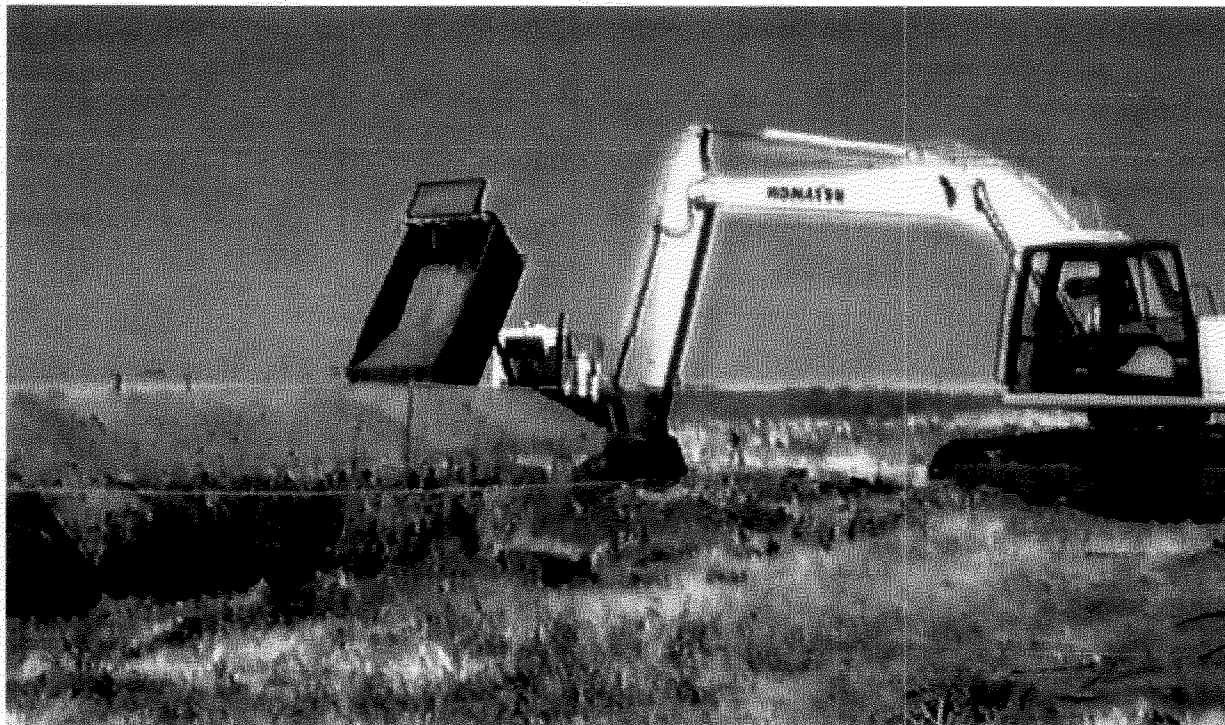


Figure 7-6. Backfilling operations.